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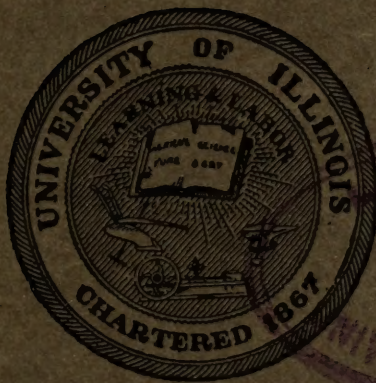
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THE UTILIZATION OF PYRITE  
OCCURRING IN ILLINOIS  
BITUMINOUS COAL

BY

E. A. HOLBROOK



CIRCULAR No. 5

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## THE UTILIZATION OF PYRITE OCCURRING IN ILLINOIS BITUMINOUS COAL

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An outline of laboratory experiments performed on a commercial scale in the mining laboratory of the University of Illinois with a view of developing a simple process for the economic recovery of the pyrite (sulphur) occurring with Illinois bituminous coal and now commonly rejected as worthless.

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### INTRODUCTION

THE mineral pyrite (or probably its true name is marcasite) occurs in nearly all Illinois coals as characteristic brassy yellow colored streaks, thin plates, lenses, nodules, bands, or balls of all sizes, sometimes up to 10 or 12 inches in thickness. In the ordinary course of mining pyrite is rejected as a deleterious impurity. Its presence in the coal in any considerable quantity not only affects the appearance and consequently the salability of the fuel, but when burned, pyrite promotes the formation of clinkers. Its heating value is small, and the sulphur dioxide gas formed from its combustion, in combination with water vapor, forms an acid which tends to corrode boiler flues and stacks.

Pyrite when pure contains more than 50 per cent of sulphur, and when the nearly pure mineral is properly burned or roasted, the sulphur dioxide gas resulting forms the basis for the manufacture of sulphuric acid. In normal times the commercial supply of sulphuric acid is obtained as a by-product of the roasting and refining of zinc, lead, or copper ores which contain sulphur. The prohibition against undue atmosphere pollution in the neighborhood of zinc, lead, or copper smelters due to the formation of sulphur dioxide and its discharge into the atmosphere, has often made its recovery a matter of necessity not regulated entirely by market demands for the resulting sulphuric acid.

In normal times in the United States, the market for pyrite has been irregular and has been limited to small areas near the points of production and utilization owing to the fact that high freight rates have operated against extensive distribution. Moreover Spanish pyrite has normally been imported easily and cheaply to the extent



of a million tons or more per year, usually in vessels which would otherwise return to America in ballast. In normal times also considerable crude sulphur has been available for the manufacture of sulphuric acid. All of these factors have affected the general market to such an extent that the commercial demand for pyrite which might be recovered during the mining and preparation of Illinois coal has been uncertain. Moreover, since the pyrite must be freed of adhering coal and refuse before shipment, the extra cost of this process has been an added disadvantage in the normal close competitive market.

In 1915, according to the report of the United States Geological Survey, 964,634 long tons of pyrite were imported while the domestic production was only 394,124 long tons, or about 29 per cent of the consumption. Of the domestic pyrite, 14,849 long tons are credited to Illinois, all of which, except a small amount from the northwestern part of the state, came as a by-product of the coal industry. Of this, possibly 12,000 tons came from one plant in the Danville district. In general then, little attention has been paid to the recovery of pyrite by the coal producers of the state.

Since the beginning of the European war new conditions have arisen. The supply of Spanish pyrite has been largely cut off, and under present market conditions (July, 1917) a clean pyrite product is worth from three to four times as much per ton as the coal with which it occurs. Sulphuric acid is used extensively in the manufacture of explosives as well as in the preparation of fertilizers, and the great increase in the demand for these products coupled with the reduction of the supply has advanced the price to a point which seems to present new possibilities in connection with the recovery of pyrite from Illinois coals. Some coals contain so large an amount of pyrite that its utilization under present conditions seems commercially practicable, and at certain mines the refuse piles contain large quantities accumulated from the picking belt at the tipple, from hand picking in the railroad cars, or from the coal washeries. It even seems probable that high sulphur coals could be crushed and washed to advantage, but with a view of saving the contained pyrite rather than of obtaining a cleaner coal. Although at present there is an almost unlimited demand for pyrite, it should be remembered that market conditions may change and, with the amount of pyrite available at most mines, large and expensive plants are probably not warranted. It becomes necessary, therefore, to design and



to install small plants which will free most of the pyrite from the coal adhering to it and will otherwise treat it to make it acceptable as a commercial product. Through the kindness of W. G. Hartshorne of Danville, the Mining Engineering Department of the University of Illinois has been able to secure several lots, each of about one ton, of crude hand-picked pyrite mixed with its adhering coal. Experimental work in the laboratories has been conducted with a view of obtaining pyrite clean enough for the market and also coal as a by-product, clean enough to pass as ordinary tippie screenings. The experiments were performed as regular class ore-dressing experiments by senior students in the department of Mining Engineering under the direction of the writer, and approved compilations from these results have been used to supply part of the data to be outlined. The possible commercial use at the present time of these results by the mining industry was suggested by F. W. DeWolf, Director of the Illinois State Geological Survey.

Attention is called to the fact that the process outlined in this circular has not been developed with the ordinary small laboratory or hand apparatus. The mining laboratory of the University of Illinois is equipped with machines which are of commercial character and which differ from commercial machines only in being in most cases of half their dimensions. It is thought, therefore, that the results of these experiments are applicable directly to a commercial plant, and it is with the hope that the information acquired may prove beneficial to the coal mining and allied industries that it is published.

## I. OCCURRENCE AND NATURE OF PYRITE IN COAL

1. *Composition of Pyrite.*—Any one who has examined lumps of bituminous coal closely or who has been underground in bituminous coal mines has observed the brassy yellow nodules, bands, or lenses which occur in the coal and which by the miners are usually called "sulphur," "sulphur balls," "cat faces," "kidney sulphur," or "brasses," depending upon the locality and upon the particular shape of the mineral. Commercially, the mineral is known as Pyrite, Pyrites, Iron Pyrites, or Iron Sulphide. When pure it contains 46.6 per cent of iron and 53.4 per cent of sulphur. The chemical



symbol is  $\text{FeS}_2$ . Another mineral of the same composition is Marcasite. This mineral has the same chemical composition as pyrite but differs from it in crystallization, and often in being a little lighter in color and in having different cleavage or breaking angles. Marcasite as a rule is more readily decomposed than pyrite and is usually of slightly less specific gravity. As a matter of fact, it is probable that most of the mineral called pyrite or sulphur and found in Illinois coal is in reality marcasite, but as the term pyrite is applied to it generally, this term will be used in this discussion.

2. *Pyrite in Illinois Coals.*—Sulphur in coal occurs in several different forms. The total sulphur content of Illinois coal ranges from 1 to 6 per cent. Of this a small amount, probably from  $\frac{1}{4}$  to  $\frac{3}{4}$  per cent of the coal appears in some organic combination which is not well understood—probably with the hydrogen and carbon of the coal. Such organic sulphur is supposedly burned completely in the process of combustion. This form of sulphur is present in all coals and is so intimately combined in the coal substance that it is not apparent to the eye and cannot be mechanically separated from the coal. Such organic sulphur does not lessen the value of a coal for combustion.

A small and varying amount of sulphur in coal is combined in the white mineral occasionally seen as flakes along the partings or planes of the coal. This is calcium sulphate or gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Many Illinois coal beds contain small percentages of this impurity, and seams No. 1 and 7 in places contain considerable amounts. The presence of sulphur in this form is of chemical interest only; it usually amounts to only a small fraction of one per cent of the coal.

The third general form in which sulphur occurs in the coal is as pyrite. Pyrite often is found in the coal in thin flakes of knife-edge thickness, occurring in vertical cleavage planes. This is especially noticeable when the coal is freshly broken. This form of pyrite may amount to several per cent of the total coal, but owing to its fine state of division, its brittleness, its thinness, and its tendency to adhere to the coal substance, it cannot be separated readily from the coal. Of greater commercial value is pyrite which occurs in the coal in the form of bands, lenses, nodules, or balls ranging up to several inches in thickness and often to several feet in lateral dimension.



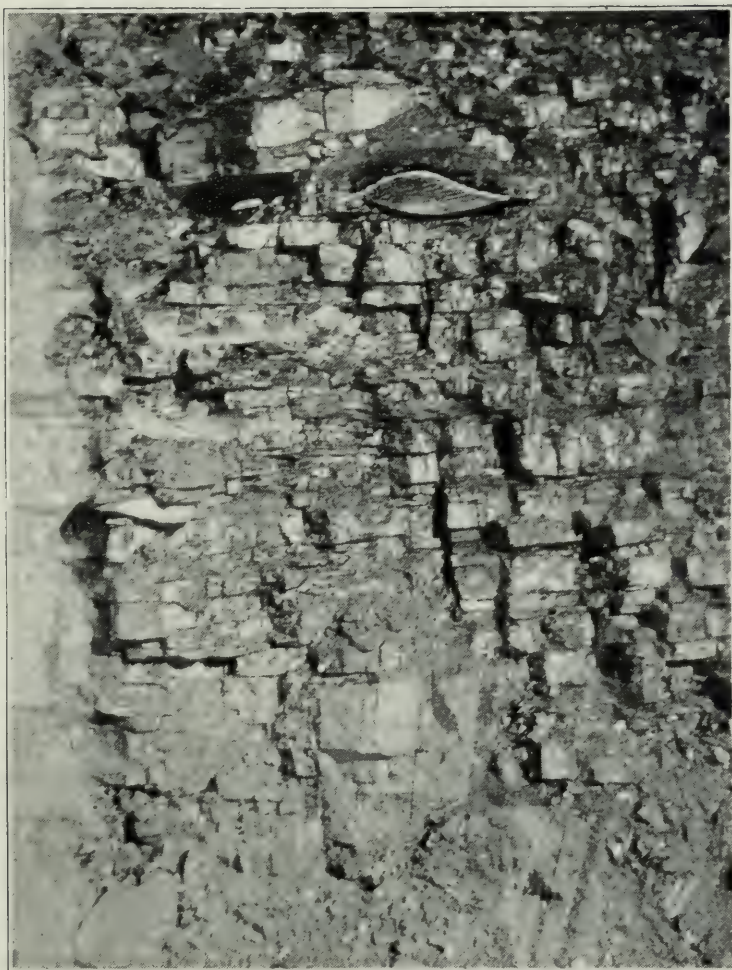


FIG. 1. PYRITE LENSE IN ILLINOIS COAL



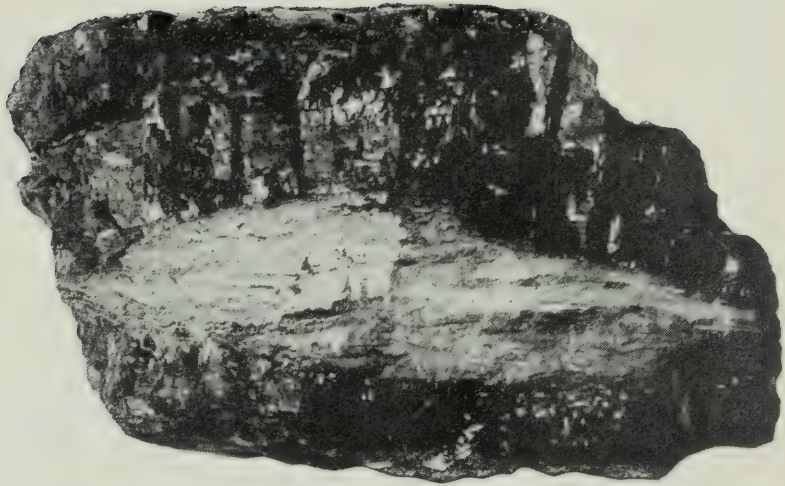


FIG. 2. CLOSE VIEW OF PYRITE LENSE IN COAL, ONE-THIRD ACTUAL SIZE

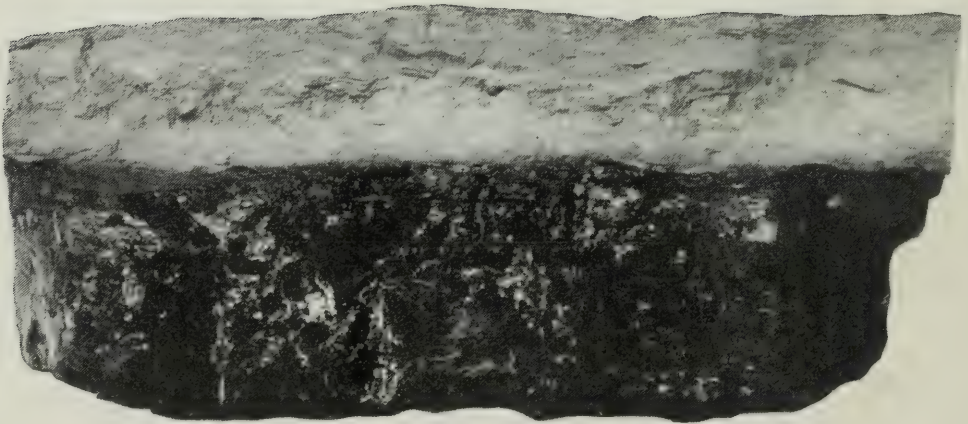


FIG. 3. CLOSE VIEW OF PYRITE BAND IN COAL, ONE-THIRD ACTUAL SIZE



Fig. 1 shows a typical pyrite lense from Illinois coal. Fig. 2 shows a closer view of a pyrite lense in coal and gives an approximate idea of the size in which it occurs. Considerable foreign carbonaceous material can be distinguished as intergrown or streaked through the lense. Fig. 3 is a close view of a typical band of nearly pure pyrite interbedded with and adhering to the coal.

On mining, the sulphur balls or bands, being brassy yellow in color and of more than three times the weight of the coal are usually distinguishable by the miner and are thrown into the waste or gob underground and discarded. Frequently pieces of pyrite are more or less covered with adhering coal and if missed by the miner may be removed by hand picking in the tippie or on the railroad cars at the surface. It does not pay to attempt to free all the coal adhering to the discarded lumps of pyrite, especially as they must be hammered or knocked off by hand. Consequently pyrite lumps derived from hand-picking the coal always have some adhering coal.

3. *Origin of Pyrite.*—The origin of pyrite in coal has been the subject of some speculation. With reference to this, it is to be noted that underground circulating waters may contain considerable amounts of iron salts, hydrogen sulphide, and gypsum and other salts in solution, which will deposit or precipitate under favorable conditions. Such conditions are furnished by the reducing tendencies of the carbonaceous matter in the coal and by the more porous layers of the seam which furnish easy channels of circulation for the solutions. The firmer bands of the seam tend to define and to limit these channels. Deposition having started around some favorable nucleus, further deposition tends to enlarge the particle. When the solution contains iron and sulphur compounds, the final result will be nodules, bands, or lenses of pyrite. Less resistance usually has been offered to the growth of these masses along the bedding or lamination planes of the coal than in other planes. For this reason pyrite bands are horizontal in the bed and may be either flat or slightly lenticular in shape. Often the bands are as much as one or two inches in vertical thickness, and they may have a horizontal area of many square feet. The lenses are sometimes 5 or 6 inches in the vertical dimension and considerably greater in the lineal dimension along the bed. Occasionally lumps of pyrite are seen, the forms of which suggest the replacement of bits of branches or other woody tissues.



That at least a part of the sulphur in coal is not due to reduction and deposition from circulating waters has been suggested editorially by *Coal Age* (Aug. 23 and Oct. 18. 1913). In general all vegetable life requires and contains the element sulphur combined in the form of sulphates. Recent analyses have shown the amount contained to be far greater than was formerly supposed. Since sulphur was probably contained in the vegetable matter forming the coal substance and since certain bacteria have the power of extracting sulphur from sulphate, it is reasonable to ascribe such a biochemical origin to at least part of the sulphur found in coal.

From this discussion it will be seen that an analysis of the total amount of sulphur in coal need not give a complete measure of the amount of pyrite which may be recovered. Those forms of sulphur which are organically combined, those which occur as gypsum, or those which occur in the very thin leaves or plates of pyrite are not recoverable. Only that form of sulphur occurring as pyrite lumps of fair size can be recovered by a washing process.

4. *Recovery of Pyrite.*—Up to the present time in Illinois only occasional efforts have been made to separate the pyrite from the adhering coal and to recover it in a condition clean enough to warrant marketing. At one mine in the Standard (Belleville) district at which seam No. 6 is worked, enough of this lump pyrite is picked in a clean condition from the coal during screening and loading to justify saving, and, in the past, shipments of the product separated by hand from the adhering coal have been made to various chemical companies for use in the manufacture of sulphuric acid. At certain mines in the Danville district, seam No. 7, numerous large bands of pyrite are hand-picked from the coal either by loaders underground or by pickers in the tipple during screening and cleaning. Several mines in this district are open-cut or stripping mines, and opportunity is thus offered for the removal of the pyrite from the coal during loading in daylight when the glistening yellow metal is easily detected.

Enough impure pyrite is being secured from several mines to justify the operation of washing, jigging, or dressing plants in which the raw pyrite with its adhering coal is crushed and washed. After the completion of this process the clean pyrite is shipped and a quantity of fairly clean coal screenings is recovered as a by-product. The



pyrite washery near Danville\* probably treats more than 100 tons of the impure pyrite with its adhering coal, per 10-hour day. Of this amount, somewhat more than 50 per cent is recovered as clean pyrite, and most of the remainder is recovered as coal.

5. *Market Conditions.*—To meet the demands of chemical companies which purchase pyrite, a certain degree of purity is necessary. Perfectly pure pyrite should contain 53.4 per cent of sulphur and 46.6 per cent of iron. Since the sulphur is the valuable component in pyrite, buyers generally specify that the finished product must contain at least 40 per cent of sulphur, and contracts are often executed on the basis of a minimum of 44 per cent. The specifications of some chemical companies also limit the maximum of carbon that may be contained in the pyrite to 8 per cent. Roughly, this means about 16 per cent of contained coal. It may be seen, therefore, that pyrite must be freed of practically all of its adhering coal in order to pass this test. The pyrite found in Illinois coal even when appearing perfectly clean does not analyze 53.4 per cent sulphur, as there is some impurity, probably carbon, generally combined or intimately mixed with it; thus a lump of apparently absolute purity would probably analyze about 50 per cent sulphur. A second requirement of the buyers in the past has been with reference to size. Lump or broken pyrite, greater than 2-inch ring size, has generally brought a higher price per unit† than pea pyrite or pyrite ranging in size from 2 inches down to about  $\frac{1}{4}$  inch or  $\frac{1}{2}$  inch, and this also has generally been worth more than the fines or pyrite below  $\frac{1}{4}$  inch. In fact the fine material has often been unsalable. Within the last year or two, specifications as to size have changed. The development of new furnaces in which the raw pyrite is burned or roasted has made fines more desirable than lump. The demand for fines has also been increased by the development of certain uses for which the iron cinder or residue from the fines can be utilized.

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\*This plant is described in an article by C. M. Young in *COAL AGE*, Vol. XI, No. 1, p. 9, Jan. 6, 1917.

† By price per unit is meant the price per per cent. of sulphur. Thus, if a pyrite contains 40 per cent of sulphur and the price is 10 cents per unit, 40 times 10, or \$4.00, is the price per ton of the material. In the same way pyrite having 45 units, or 45 per cent of sulphur is worth \$4.50 per ton. It generally pays, therefore, to prepare and ship the highest grade product, providing, of course, that in preparation, the losses of material are not greater than the benefits to be gained.



While no definite prices per ton for the different sizes can be quoted owing to the changing market conditions, information received from what are believed to be reliable sources states that the present market price (July, 1917) for pyrite is as high as 20 cents per unit, f. o. b. point of production, and the belief is expressed that the price will not be lower than 15 cents per unit during the next two years. This compares with a former price of 10 cents per unit ruling two years ago. Twenty cents per unit equals \$8.00 per ton if the pyrite contains 40 per cent of sulphur, or more than four times the value per ton of the coal at the mine.

There are many mines in Illinois at which considerable pyrite is thrown into waste or gob underground, or is discarded at the picking belt at the surface. Through the agency of the Illinois Co-operative Coal Mining Investigations many analyses are available showing the amount of sulphur contained in Illinois coals. These indicate that face samples taken underground have a sulphur content ranging from less than 1 per cent to more than 6 per cent, of which amount probably 60 to 90 per cent is combined in the form of pyrite. The directions given the samplers in this work called for "the exclusion of sulphur bands or balls above  $\frac{3}{8}$  inch in thickness or of thinner ones if, in the judgment of the sampler, these are excluded by the miner in loading his coal." It is evident that these "excluded" bands comprise the important product from the standpoint of pyrite recovery. For this reason no sulphur analyses of face samples are presented herein.

Considerable quantities of pyrite might be recovered in many parts of the state, particularly in seams Nos. 1 and 2 in the north-western part, in the LaSalle district where seam No. 5, or the "second vein" coal, is worked, in seam No. 6 in the Belleville and Staunton districts, in seam No. 5 in the Harrisburg district and in seam No. 7, where mined (usually in the Danville district). At the present time the demand is so great that some mines are shipping the hand-picked pyrite without further cleaning, even though its content of sulphur is not so high as 40 per cent. Chemical companies, however, are buying this material at a reduced price per ton.

At the present time in Illinois there are about twenty-five coal washeries producing clean coal. The refuse from these washeries contains from about 4 to more than 22 per cent of sulphur, generally in the form of pyrite lumps.



In contrast with some pyrite derived from other sources, the pyrite from Illinois coal contains no arsenic or antimony, impurities which sometimes impair an otherwise salable pyrite. Illinois pyrite also burns and decomposes easily. On the other hand, any considerable percentage of carbon or coal in the material causes the gas to be smoky and the resultant acid to be dirty. Modern methods of scrubbing or cleaning the gas have partially overcome this difficulty. The tendency of the Illinois pyrite to oxidize and decompose readily is a factor which does not permit of storage for any considerable period. Certain pyrite from other sources contains some copper which may be recovered after burning, thus adding to its value per ton.

6. *Pyrite in Refuse Heaps.*—Table 1 shows the percentages of sulphur in several of the waste heaps at Illinois washeries, and also that in the refuse from several tippie picking belts. Some of the washeries have been running for years and have accumulated large heaps or dumps of the refuse. The possibility that these might profitably be retreated at the present time to recover their pyrite content is worthy of consideration. Should the present market continue, the probability of profitably washing high pyrite coals with a view of producing pyrite rather than of benefiting by the added value of the washed coal can be given serious attention. It is a fact that certain mines in Illinois have been abandoned owing to the high pyrite content of the coal. The possibility of reopening such mines as pyrite producers, at the same time recovering the coal as a by-product, is a subject which can be considered at this time.

7. *Oxidation of Pyrite.*—The pyrite obtained from Illinois coal oxidizes or weathers very rapidly on exposure to the air. Within a few days after mining, a white salt may be noticed on the surface of the lumps. This white salt is iron sulphate,  $\text{FeSO}_4$ , and is the product of the absorption by the pyrite of oxygen from the air. Thus, if exposed for any length of time, the pyrite tends, at least on the surface, to become oxidized and to the extent of such oxidation to be rendered valueless. Pyrite masses in heaps which have the appearance of being oxidized, if examined closely, will often be found to have been affected only at the surface, and what may seem to be a mass of



TABLE 1<sup>1</sup>

AMOUNT OF SULPHUR AND PYRITE IN REFUSE OF COAL AT VARIOUS  
ILLINOIS COAL PREPARATION PLANTS

| Seam No. | Plant      | Situation      | Character of Material              | Percentage of Sulphur                                 | Percentage of Pyrite in Material <sup>2</sup> |
|----------|------------|----------------|------------------------------------|-------------------------------------------------------|-----------------------------------------------|
| 2        | Washery    | Northern Field | Washery Refuse                     | 9.35                                                  | 16.83                                         |
| 2        | Washery    | Northern Field | Washery Refuse                     | 22.01                                                 | 39.62                                         |
| 2        | Washery    | Northern Field | Washery Refuse                     | 8.06                                                  | 14.51                                         |
| 2        | Washery    | Northern Field | Washery Refuse                     | 4.57                                                  | 8.14                                          |
| 2        | Washery    | Northern Field | Washery Refuse                     | 5.27                                                  | 9.49                                          |
| 2        | Washery    | Northern Field | Washery Refuse                     | 6.77                                                  | 12.19                                         |
| 2        | Washery    | Northern Field | Washery Refuse                     | 4.21                                                  | 7.58                                          |
| 6        | Washery    | Central Field  | Washery Refuse                     | 16.55                                                 | 29.79                                         |
| 6        | Washery    | Central Field  | Washery Refuse                     | 12.57                                                 | 22.63                                         |
| 6        | Washery    | Central Field  | Washery Refuse                     | 22.45                                                 | 40.41                                         |
| 6        | Washery    | Southern Field | Washery Refuse                     | 10.42                                                 | 18.76                                         |
| 6        | Washery    | Southern Field | Washery Refuse                     | 4.34                                                  | 7.81                                          |
| 6        | Washery    | Southern Field | Washery Refuse                     | 13.67                                                 | 24.61                                         |
| 6        | Washery    | Southern Field | Washery Refuse                     | 10.03                                                 | 18.05                                         |
| 6        | Washery    | Southern Field | Washery Coarse Refuse              | 10.25                                                 | 18.45                                         |
| 6        | Washery    | Southern Field | Washery Fine Refuse                | 10.03                                                 | 18.05                                         |
| 6        | Tipple     | Southern Field | Picking Belt Refuse<br>Hand-Picked | 4.84                                                  | 8.71                                          |
| 6        | Rescreener | Southern Field | Hand-Picked                        | 6.60                                                  | 11.88                                         |
| 5        | Tipple     | Southern Field | Hand-Picked                        | 12.00                                                 | 21.60                                         |
| 7        | Mine       | Danville       | Hand-Picked                        | 50 tons crude pyrite hand-picked from 1,000 tons coal |                                               |

<sup>1</sup> Washery results from "Coal Washing in Illinois," by F. C. Lincoln, Univ. of Ill. Eng. Exp. Sta., Bul. 69, 1914.

<sup>2</sup> Assuming 90 per cent of sulphur is in pyrite form and that this is 50 per cent sulphur.

powdery white iron sulphate will in reality be found to be a lump of solid pyrite with only a coating of the sulphate. This coating ( $\text{FeSO}_4$ ) especially when freshly formed is soluble in water and should be easily removed during the washing or cleaning process.

Washed pyrite when stored immediately may heat slightly while drying. Experience has shown, however, that pyrite will not fire if it is comparatively pure and does not contain considerable coal, bits of wood, or other easily ignitable material.

8. *Description of Machinery used in Preparing Pyrite and Outline of Washing Tests.*—Owing to the uncertainty of the market conditions, any installation for the purpose of recovering pyrite, especially if only a few tons per day of mixed pyrite and coal are to be recovered, must be simple in character, of low cost, and capable of being operated by unskilled labor. It is believed possible for several mines in a district to co-operate, if necessary, in the erection of a small plant of a character similar to the one hereinafter described.

Since the hand-picked pieces of pyrite from coal range up to several inches thick and more than a foot square, and since pieces of coal tend to adhere to the lumps, hand-picking in general will not produce a high grade product. It is true that by hand-picking and hammering the larger lumps may be freed of coal sufficiently to produce a salable product, but this method involves the waste of the large amount of pyrite which occurs in pieces smaller than 2 inches in diameter, or of a size too small to permit hand-picking to be done profitably. It should be remembered also that the fine pyrite is of greater value per ton than the coarse material.

Since the specific gravity of the pyrite is high (4.7 to 5.1) as compared with that of coal (1.3), washing by a process involving jigging or agitation in water causes the heavy mineral to sink rapidly while the light material may be drawn off at the top. This principle of separation is used in the ordinary jig.

With the purpose of devising some simple washing or ore-dressing process to effect a separation of the pyrite from its adhering coal, the Department of Mining Engineering of the University of Illinois has undertaken a series of tests with various samples of pyrite. As a result of these experiments an arrangement of machines has been worked out, and the power required and the cost of operation have been determined for a simple plant capable of preparing nearly pure pyrite on the one hand and commercial coal on the other.

The mining laboratory at the University of Illinois is equipped with rock crushers, breakers, and rolls of several different kinds installed in such manner as to make possible the determination of the best method or crushing any ore or coal to the size necessary for subsequent treatment. With this equipment are screens of the revolving or trommel type, and shaking and vibrating screens to divide the crushed material into the several sizes required for further treatment. There are also jigs of the plunger, Harz, or Luhrig type.



and jigs of the basket or Stewart type. These jigs separate the valuable mineral from the refuse. In addition special machines in the form of concentrating tables are installed for special treatment of fine or small material, that is, material too small to be successfully handled by jigs.

Preliminary tests were made by crushing the crude pyrite to various sizes in different types of crushing machinery such as breakers, rolls, and pulverizers, and by comparing the various samples of pyrite to determine the extent to which separation of pyrite and coal had been effected. Sizing tests were made on various kinds of screens and separation of these products was effected by different types of jigs, washers, and concentrating tables. The Delamater float and sink test machine was particularly useful in determining roughly the possibilities of separation of various sizes of mixed coal and pyrite. The possibility of separating pyrite from coal by a strong electric magnet was also tried, but under the influence of a 6-ampere 40-ohm electric magnet installed in a Dings electromagnetic separator, the results were negative. Without describing in detail the various tests, it is sufficient to give an outline and to present the average results of those tests which proved most successful, and which the writer is convinced give a high percentage of recovery at a low or reasonable cost.

## II. SUMMARY OF TESTS

9. *Machinery Required.*—The tests performed lead to the conclusion that the practical separation of pyrite from Illinois coal for the purpose of obtaining a commercial grade of pyrite, with coal as a by-product, presents no difficulty when performed by crushers, screens, and concentrating machines adapted to ordinary ore-dressing work. The chief problem is to secure a plant of the greatest simplicity and of the lowest cost. At the same time it should be of good capacity and should yield a high percentage of recovery of the pyrite.

10. *Percentage of Recovery.*—The experiments from which the data are to be detailed indicate that a simple plant will recover about 81 per cent of the pyrite in the coal, and that if the middlings product from the jig is crushed and retreated, this recovery can be increased

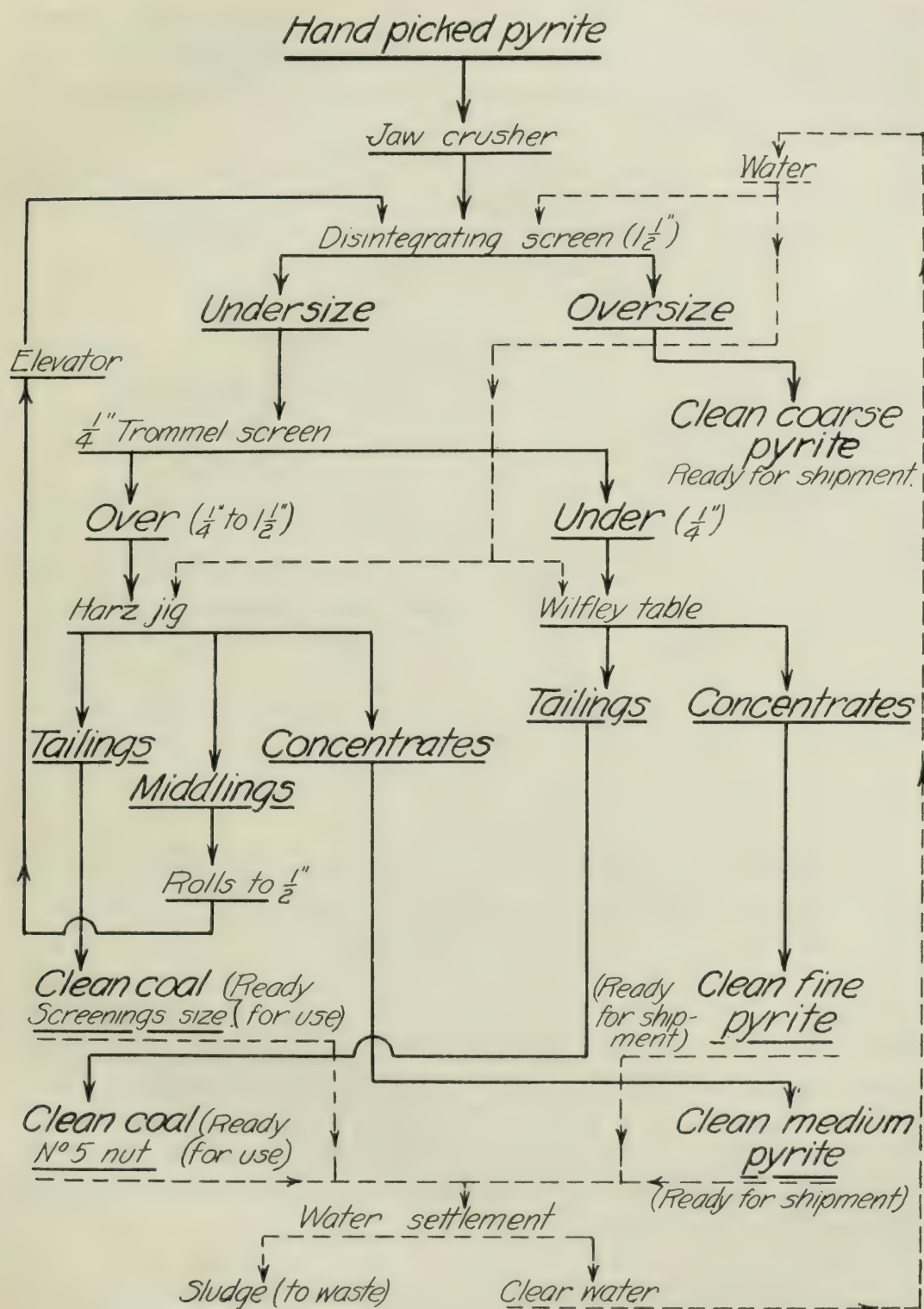


FIG. 4. SIMPLIFIED FLOW SHEET, PYRITE-COAL CONCENTRATION



to about 87 per cent. This pyrite will average more than 40 per cent of sulphur and may be sold directly to chemical or to fertilizer companies. The coal recovered as a by-product is not greatly inferior to ordinary screenings.

TABLE 2  
ESTIMATED OPERATING STATEMENT OF A PYRITE PLANT OF A  
CAPACITY OF 50 TONS PER 8-HOUR DAY

| DEBIT                                                                                                   | CREDIT                                                                                                       |
|---------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|
| 50 tons of Hand-Picked Pyrite at<br>\$1.35 . . . . . \$ 77.50                                           | Coarse Pyrite 24,000 lb., 45 per cent<br>Sulphur at 15 cents per unit = \$6.75<br>per ton . . . . . \$ 81.00 |
| Interest and Depreciation on Plant<br>Investment of \$18,000 at 20 per<br>cent per year . . . . . 12.00 | Pea Pyrite 22,000 lb., 45 per cent Sul-<br>phur at 15 cents per unit . . . . 74.25                           |
| Labor, 5 men at \$3.00 . . . . . 15.00                                                                  | Fines 6,510 lb., 41 per cent Sulphur at<br>15 cents per unit . . . . . 20.02                                 |
| Supplies and Renewals . . . . . 15.00                                                                   | Extra Pyrite if Middlings are retreated<br>=4,290 lb., 43.5 per cent Sulphur . . 13.99                       |
| Power, 50 H.P. . . . . 10.00                                                                            | Coal 35,231 lb. at \$1.00 per ton . . 17.62                                                                  |
| \$129.50                                                                                                | Loss (allowing for coal in middlings<br>as loss) 8,325 lb. . . . .                                           |
|                                                                                                         | \$206.88<br>129.50                                                                                           |
|                                                                                                         | Profit per day . . . . . \$ 77.38                                                                            |
|                                                                                                         | Profit per ton of raw pyrite . . . \$ 1.55                                                                   |

11. *Estimated Operating Result.*—An effort has been made to forecast the results of operating a 50-ton per 8-hour day pyrite plant, under conditions comparable to the average to be met at Illinois mines where pyrite is to be found in sufficient quantities to warrant recovery. The summary presented in Table 2 is based partly upon estimated figures, especially with reference to the cost of crude pyrite as laid down at the plant. The figures given for the value of the product are based on a price of 15 cents per unit of sulphur. The capital cost of the plant will be found detailed on page 40. In nearly every case, the estimates are believed to represent maximum costs and conservative selling prices.

12. *Method of Operation.*—Fig. 4 is a diagrammatic illustration or simplified flow sheet of the treatment plant recommended as a result of the tests performed. The successive steps believed essential

to the complete treatment of such pyrite are shown. Fig. 5 shows the same flow sheet with percentages of recovery attained at each part of the process. This indicates results which might be accomplished in practical work. Owing to the difficulty experienced in regulating machines for the relatively small tonnage treated in laboratory work, it is believed that in every case, commercial practice on a large scale

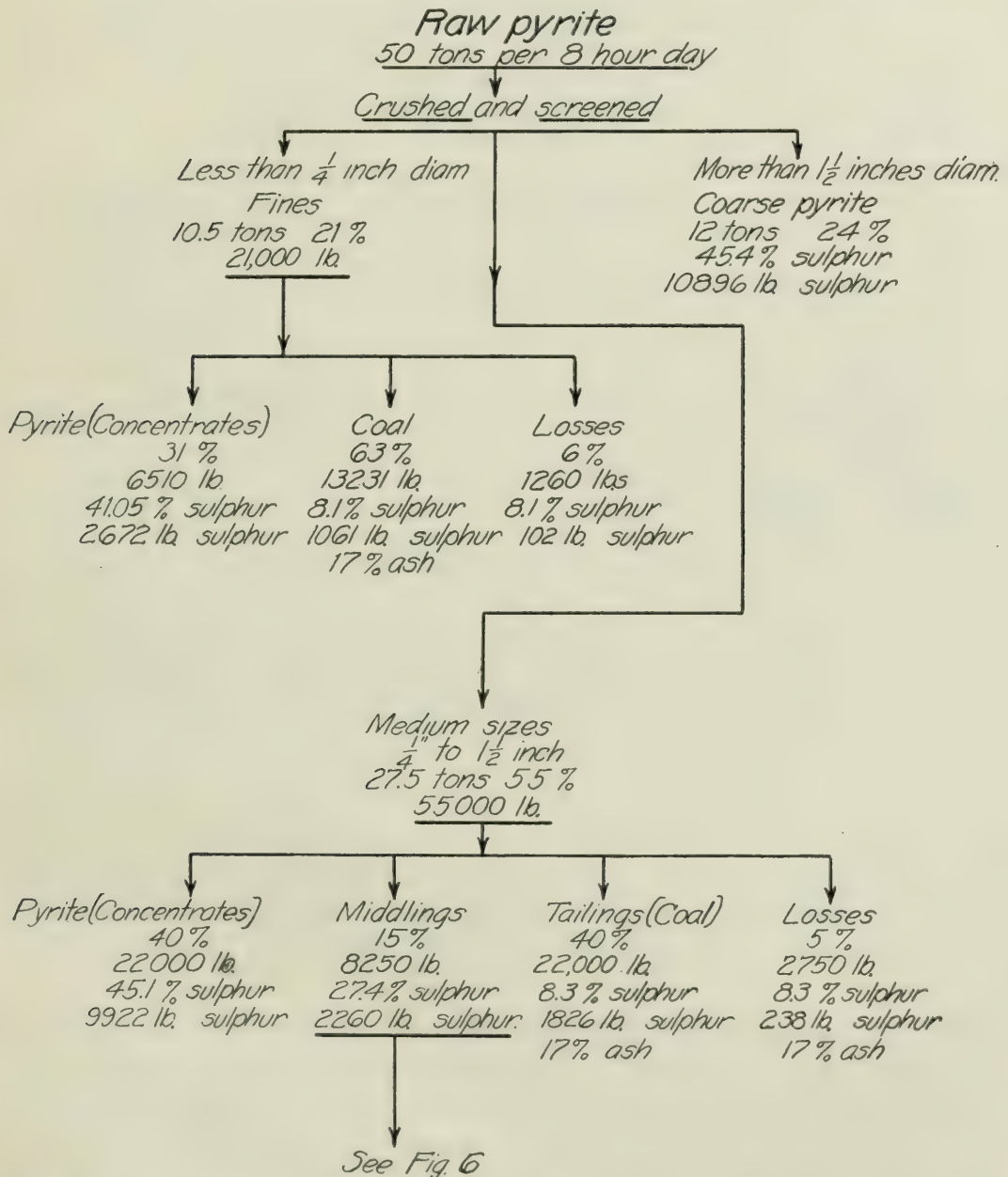


FIG. 5. OUTLINE OF TREATMENT OF RAW PYRITE SHOWING BALANCE OF VARIOUS PRODUCTS AND ACCOUNTING FOR SULPHUR



would result in higher recovery than is indicated by this outline. The tonnage is based on a plant capable of treating 50 tons of crude hand-picked pyrite per 8-hour day, as this is believed to represent the largest plant needed by one mine or even by several mines combined.

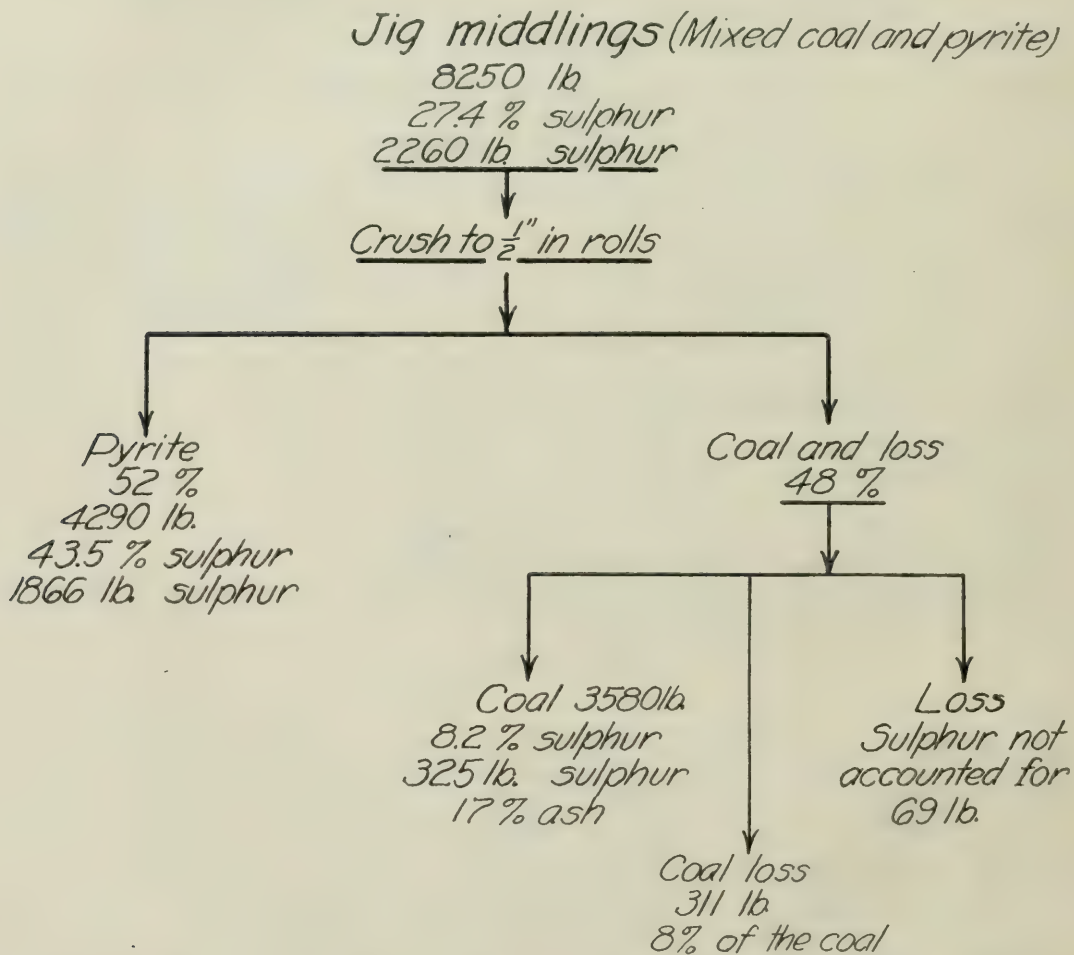


FIG. 6. OUTLINE OF FURTHER TREATMENT OF JIG MIDDINGS, SHOWING BALANCE OF PRODUCTS

Fig. 6 is a diagram which indicates the possibilities of recrushing the middlings product obtained from the second compartment of the jig, then screening it through the  $\frac{1}{4}$  inch trommel screen, and allowing it to pass either to the jig or to the concentrating table, according to its size. In this way the recovery can be increased by about 6.4 per cent.

Table 3 shows the amount and percentage of recovery or loss of the pyrite in each operation of the process, based on sulphur content as determined by sampling and by analysis of each of the products recovered.

TABLE 3  
FLOW SHEET REDUCED TO A BASIS OF SULPHUR CONTENT, SHOWING THE AMOUNT OF SULPHUR IN EACH PRODUCT BASED ON THE OUTPUT OF A PLANT HAVING A CAPACITY OF 50 TONS PER 8-HOUR DAY

| Product                            | Name           | Size               | Sulphur Per Cent | Sulphur Content Lb. <sup>1</sup> | Sulphur Recovered Lb. | Per Cent of Total Sulphur Recovered or Lost |
|------------------------------------|----------------|--------------------|------------------|----------------------------------|-----------------------|---------------------------------------------|
| Coarse (Screen) Concentrates       | Lump Pyrite    | Above 1 ½ in. ring | 45.4             | 10896                            | 10896                 | 37.6                                        |
| Fine (Table) Concentrates          | Fine Pyrite    | Under ¼ in.        | 41.05            | 2672                             | 2672                  | 9.2                                         |
| Medium (Jig) Concentrates          | Pea Pyrite     | 1 ½ in. to ¼ in.   | 45.1             | 9922                             | 9922                  | 34.2                                        |
| Total                              |                |                    |                  |                                  | 23490                 | 81.0 Recovery                               |
| Middlings from Medium Concentrates |                | Crushed to ½ in.   |                  | 2260                             | 1866                  |                                             |
| Total with Middlings Added         |                |                    |                  |                                  | 25356                 | 87.4 Recovery                               |
| Fine Coal                          | No. 5 Nut      | Under ¼ in.        | 8.1              | 1826                             |                       |                                             |
| Coal from Jig Overflow             | Screening Size | 1 ½ in. to ¼ in.   | 8.3              | 1061                             |                       |                                             |
| Loss (Jig)                         | Coal           | Estimated          | 8.3              | 102                              |                       |                                             |
| Loss (Table)                       | Coal           | Estimated          | 8.1              | 238                              |                       |                                             |
| Loss (Middlings)                   | Pyrite         | Not acct. for      | 69 lb.           |                                  |                       |                                             |
| Total Sulphur in Original Product  |                |                    |                  | 28977                            |                       |                                             |

<sup>1</sup> 28,977 pounds of sulphur from 50 tons of material amounts to 28.98 per cent of sulphur in original crude hand-picked pyrite (assuming all sulphur to be in the form of pyrite). For pyrite containing 53.4 per cent of sulphur, the total pyrite content of the crude pyrite would be 54,264 pounds, or 54.26 per cent of pyrite, and, therefore, the content of coal and contained ash and shale would be 45.74 per cent. The total recovery from disintegrating screen, jig, and table on the crushed crude pyrite was 81.0 per cent of the total pyrite or 23,490 pounds of sulphur from the 28,977 pounds contained in 100,000 pounds of crude pyrite. If the middling product from the jig is recrushed and treated, the recovery is increased to 87.4 per cent, or 25,356 pounds.

The coal produced as a by-product contains about 8 per cent of sulphur, a part of which is in the form of pyrite. In commercial operations extending over considerable periods this loss of pyrite could be decreased, as it is largest when starting and while shutting down the machinery. These operations occur frequently in experimental runs. The amount of coal recovered as a by-product is



considerable, the tests indicating 38,811 lb. per day from the plant and product under discussion, or from 18 to 20 tons. It should be remembered that this coal is of screening size, and that its purity depends largely upon the care with which the pyrite is removed during the process of cleaning.

13. *The Tests and the Results.*—In the final tests the pyrite as received (about a ton in weight) contained from 25 to 28 per cent of sulphur, or about 50 per cent by weight of pyrite. The other 50 per cent of the mineral consisted of coal adhering to the lumps and intermixed with the bands of pyrite. The material had been hand-picked at a tippie preparing No. 7 coal in the Danville district. The lumps including the adhering coal were as large as 6 or 8 inches in thickness, and were often a square foot in area. This material was first put through an ordinary rock breaker. The rock breaker in the laboratory is of the Gates gyratory type (Fig. 7), but from tests made with a Blake type of rock breaker it is believed that the latter type will be equally satisfactory and probably cheaper in first cost. Attention is here called to the fact that ordinary coal crushing machinery is not suitable for crushing raw pyrite. The pyrite is extremely hard and only breakers designed for hard rock should be used. Breakers designed for soft material do not possess adequate strength, and the wear will be excessive if used on this class of material.

The breaker was set with a throat opening, or discharge, about  $1\frac{1}{2}$  inches wide, and although the pieces discharged through this had a thickness of not more than  $1\frac{1}{2}$  inches, the area of some of the lumps was several square inches in extent owing to the tendency of the pyrite to break into flat slabs. Examination showed that this breaking process caused a large portion of the lump pyrite to separate from the adhering coal. The coal itself tended to break into cubical pieces. Also the coal, because of its brittleness, generally broke up into finer sizes than the pyrite. After breaking, the large lumps of pyrite had only small bits of coal adhering to them. Thus it was decided to screen this material in an attempt to secure a coarse pyrite which would be sufficiently clean for the market.

The crushed material was put through a revolving or trommel screen having round hole openings of about the same diameter as the opening in the rock breaker. Fig. 8 illustrates the housing

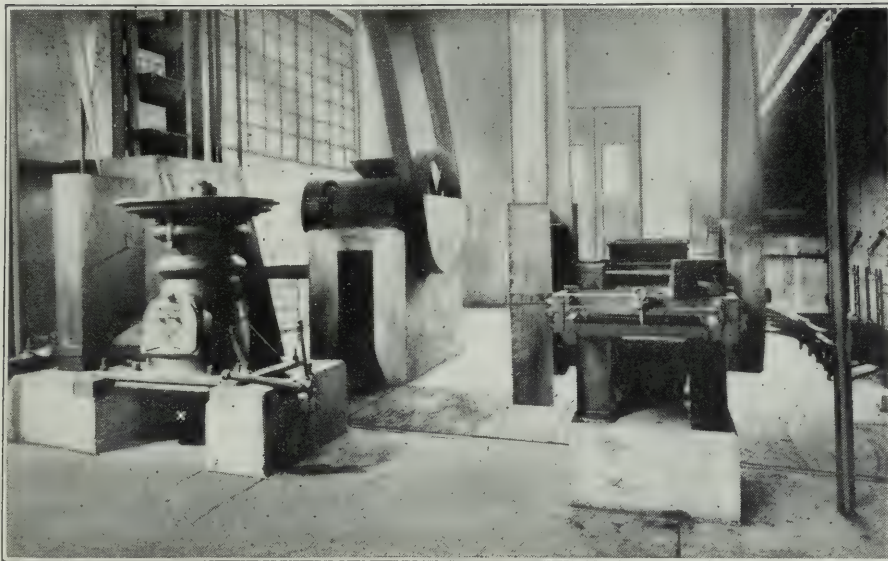


FIG. 7. GYRATORY ROCK BREAKER IN UNIVERSITY OF ILLINOIS MINING LABORATORY  
(MACHINE MARKED WITH A CROSS)



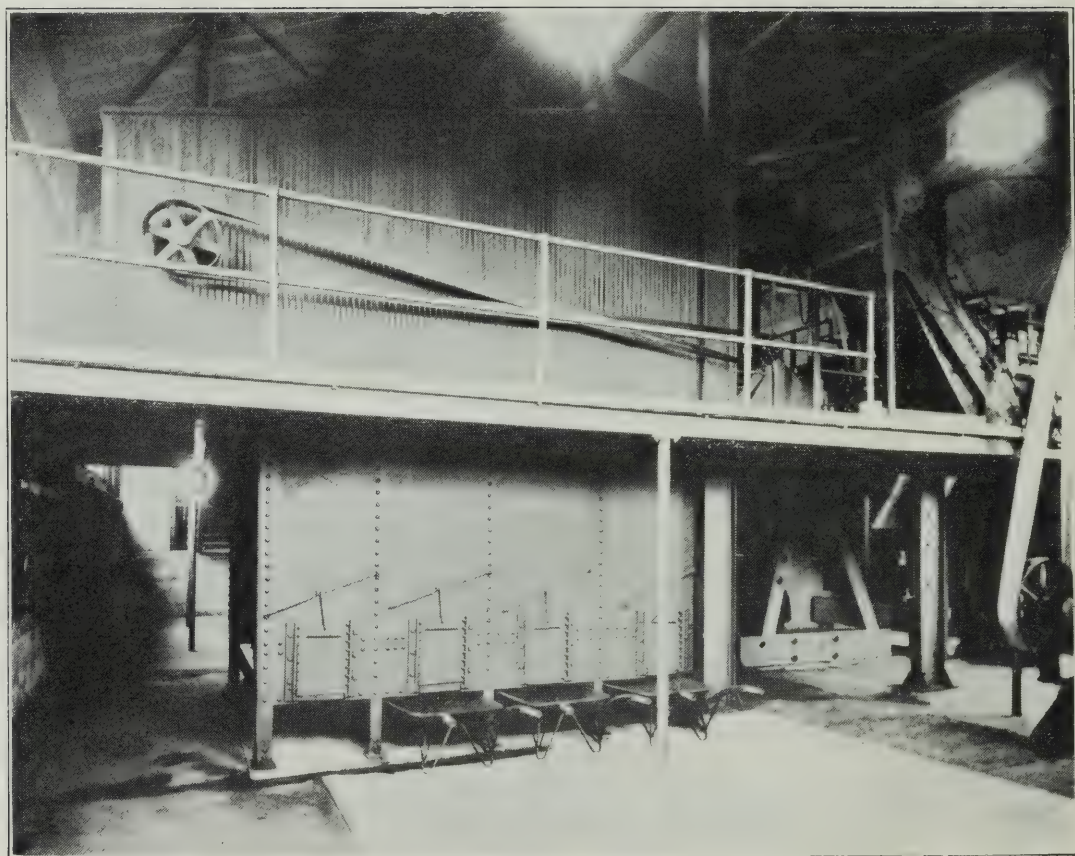


FIG. 8. HOUSING IN WHICH REVOLVING SCREEN IS CONTAINED, WITH BINS UNDERNEATH. UNIVERSITY OF ILLINOIS MINING LABORATORY

in which the revolving screen used in the experiments is contained, and shows it to be of commercial size. Since the coal tended to break into cubical pieces while the pyrite tended to break into flat pieces, it was thought that a separation could be made of the two by simple screening alone. This expectation was borne out by results obtained. Later, steel lifters were introduced in the revolving screen as shown in Fig. 9. During screening these lifters caused the material to be carried to the top of the screen and to be dropped several feet. The impact from this fall served to break any large coal so that it passed through the screen, and it also freed the pyrite of any small particles of adhering coal. It was shown also that lump pyrite may, if desired, be further cleaned by screening the material while wet, that is, by introducing sprays of water into the screen. The rubbing action of the wet material against the screen serves to loosen most of the specks of coal remaining on the coarse pyrite so that they may pass through the screen. The greater the diameter of the screen, that is, the greater the length of fall of the particles after having been lifted, the freer is the oversize, or clean coarse pyrite of coal impurity.

14. *The Size of Screen Holes.*—As previously mentioned the largest size of screen opening was about a  $1\frac{1}{2}$  inch round hole. The screen illustrated in Fig. 9 is not unlike the Bradford disintegrator which is in common use for cleaning coal to free it of lumps of shale, pyrite, sticks of wood, bits of iron, and other impurities. The result of this screening was the production of 21 per cent of the total amount treated as clean lump pyrite of  $1\frac{1}{2}$  inches in minimum size, and of an analyzed purity which in all the tests ran more than 40 per cent sulphur and in some as high as 45.4 per cent. By this simple process of crushing and screening, it was possible to produce 37.6 per cent of the pyrite immediately in the form of a clean marketable product.

The material passing through the  $1\frac{1}{2}$  inch holes of the disintegrating screen entered a small trommel or revolving screen having a screen plate with holes about  $\frac{1}{4}$  inch in diameter. The purpose was to separate the material smaller than  $1\frac{1}{2}$  inches into two sizes, one of which should contain all sizes between  $1\frac{1}{2}$  inches and  $\frac{1}{4}$  inch, and the other, all sizes below  $\frac{1}{4}$  inch. If desired, the same results could be obtained by adding an outer screen plate with  $\frac{1}{4}$  inch round holes to the disintegrating screen, that is, by making it a compound concentric screen. In the writer's opinion, it is more satisfactory



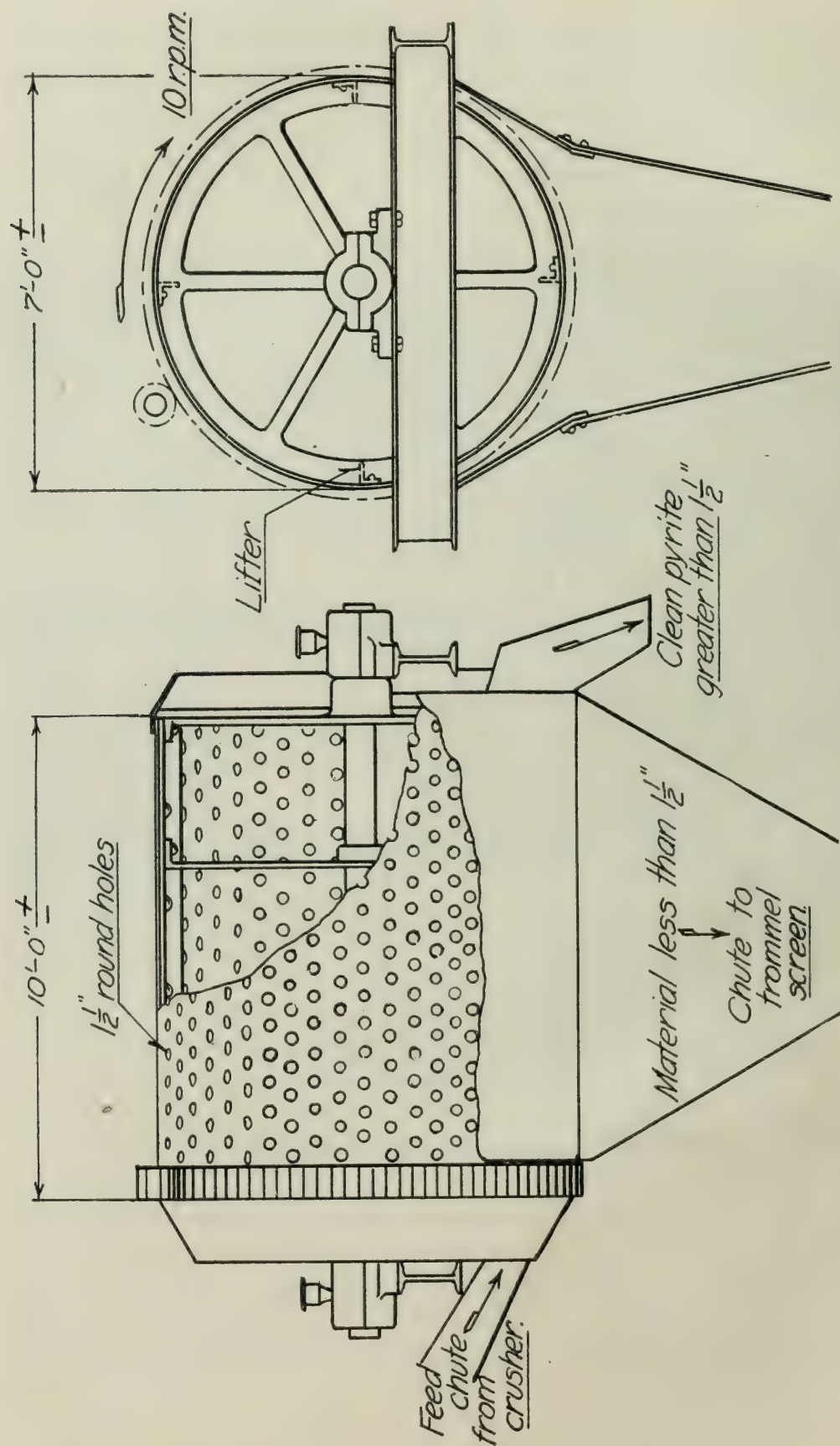


FIG. 9. TROMMEL SCREEN WITH LIFTERS, EQUIPPED AS A DISINTEGRATING SCREEN

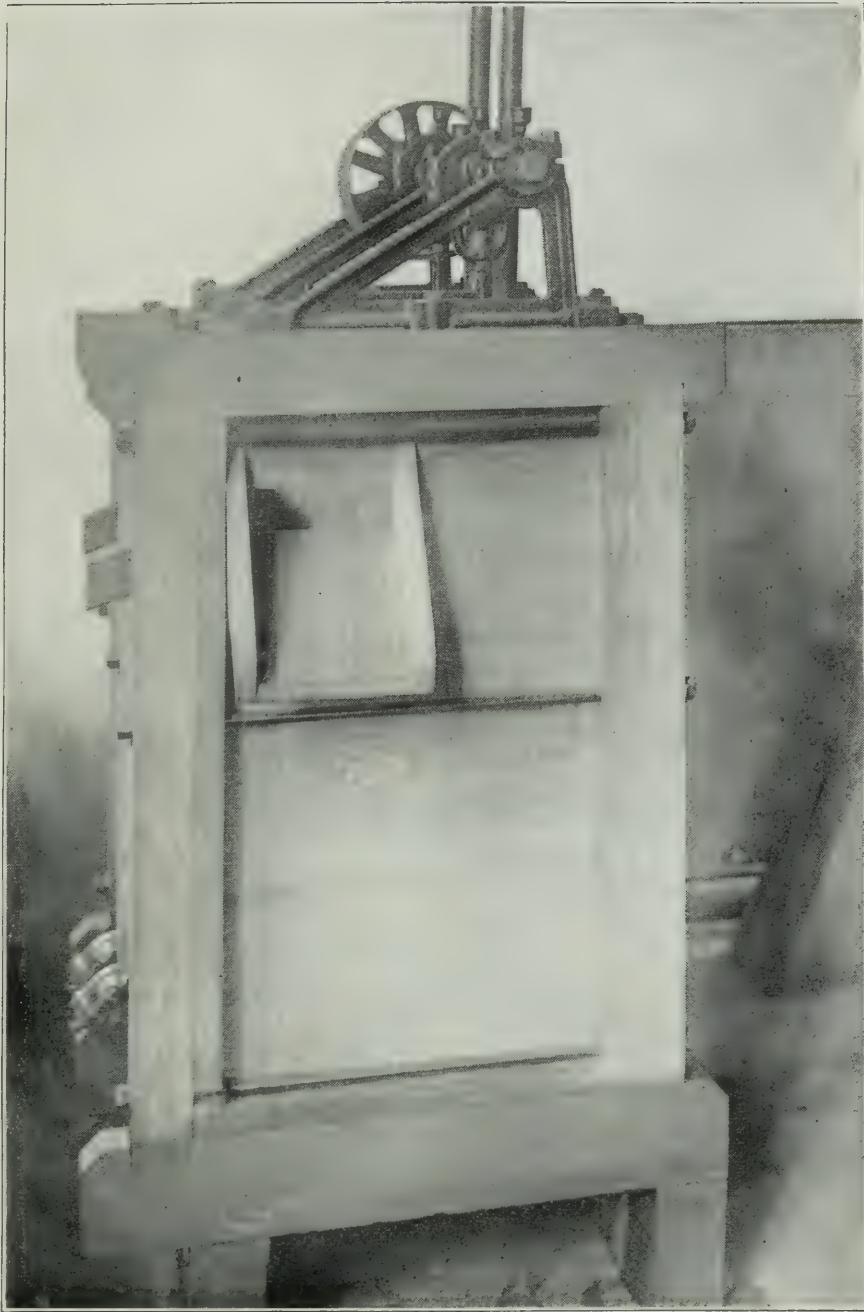


FIG. 10. HARZ OR LUHRIG PLUNGER JIG. UNIVERSITY OF ILLINOIS MINING  
LABORATORY



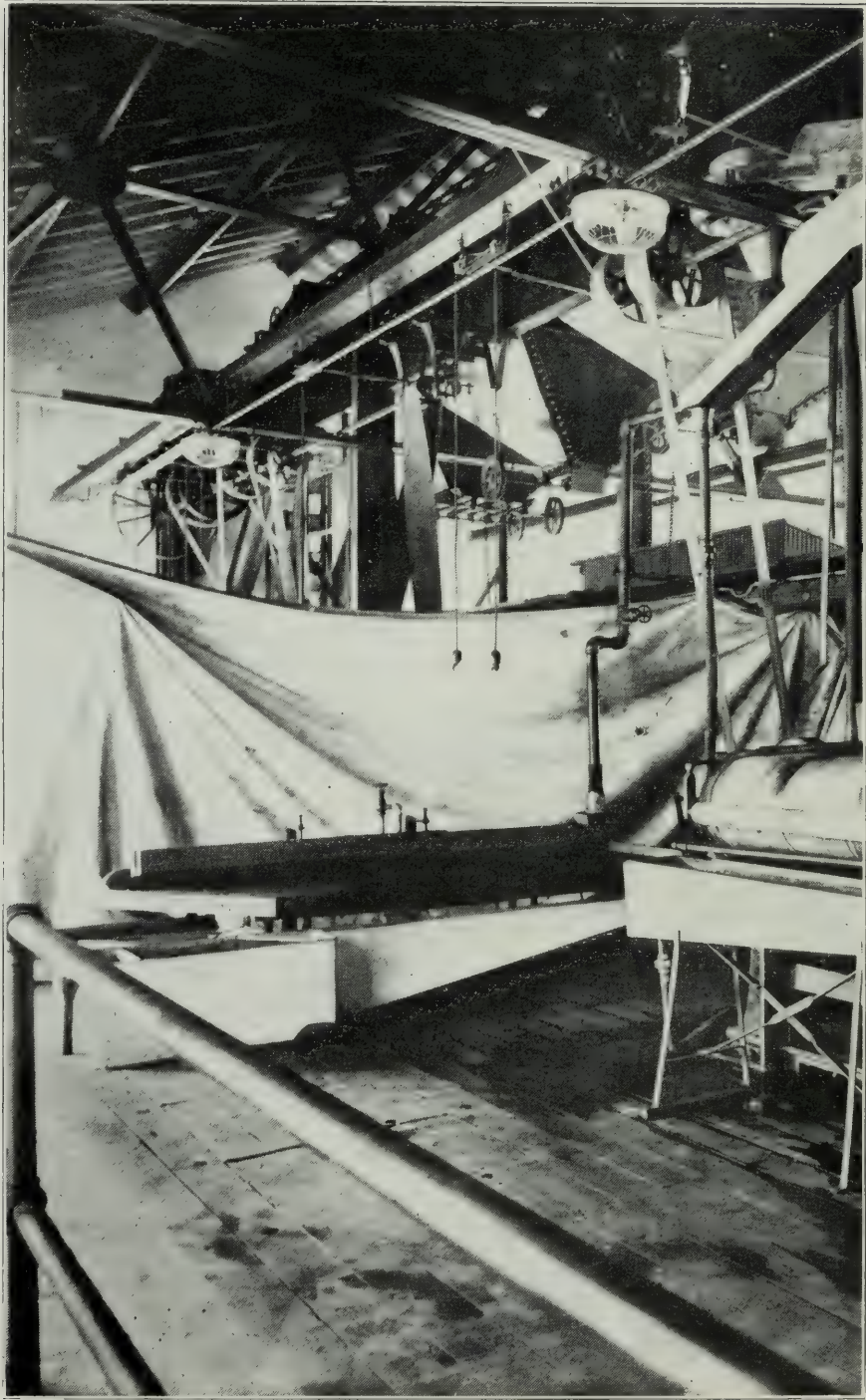


FIG. 11. LABORATORY ORE CONCENTRATING TABLE, ONE-HALF COMMERCIAL DIMENSIONS

to use separate screens, especially if the matter of making repairs easily is considered. Where all the sizes less than  $1\frac{1}{2}$  inches in diameter were washed or jigged together, the separation of the pyrite from the coal was incomplete, especially in the fine sizes below about  $\frac{1}{4}$  inch. Jigs are not well adapted for the treatment of these fine sizes, therefore separate treatment of the material below  $\frac{1}{4}$  inch should be made on a special concentrating table designed for fine material.

Of the amount falling through the holes of the disintegrating screen, 70 per cent was larger than  $\frac{1}{4}$  inch. This material larger than  $\frac{1}{4}$  inch in diameter and smaller than  $1\frac{1}{2}$  inches was sent to a two-compartment Harz or Luhrig plunger jig. The jig used in the laboratory (Fig. 10) is of the two-compartment commercial type and is of half dimensions, capable in every way of giving commercial products. From this jig three products were obtained: Number 1 was a clean pyrite product from the first compartment draw-off which amounted to 22 per cent of the total feed or 34.2 per cent of the total pyrite in the mineral. The sulphur content of this product ranged from 42 per cent to 46 per cent. Number 2 was material from the second compartment amounting to 7.8 per cent of the total pyrite or 2.3 per cent of the amount fed. This material was a true middling product; that is, it consisted of pieces of pyrite and coal which had not been freed from each other. In other words, the weight of any piece lodging here was not quite sufficient to cause it to settle in the first compartment, and still the piece was not light enough to allow it to overflow the second compartment. Number 3 was the overflow material from the second compartment which was found to be practically clean coal. In the preliminary runs some pieces of pyrite were observed in this clean coal, but after a few trials to get the correct adjustment of the jig, no difficulty was experienced in obtaining a coal comparable with the ordinary screenings furnished by Illinois coal mines.

Middlings such as were noted in the second compartment were not in condition to be marketed since their sulphur content was only 27.4 per cent. In commercial practice, if the quantity of these middlings is sufficient to warrant it, more nearly complete separation may be accomplished by recrushing to a finer size and passing again through the disintegrating screen.

In coal washing work in Illinois little attention has been paid to material under  $\frac{1}{4}$  inch, largely because material of this size usually



contains an excess of refuse and because it does not readily dry out or free itself of water. In pyrite washing, however, conditions are different. A considerable portion (21 per cent) of the material crushed in the rock breaker will be found to be under  $\frac{1}{4}$  inch in size. Since this material contains about 42.2 per cent of pyrite and since this fine pyrite has become more valuable than the larger sizes, some form of modern ore-concentrating table should be used to separate pyrite from coal in these sizes. No difficulty will be found in freeing these sizes of pyrite of water. In the experimental work, a laboratory concentrating table of half commercial dimensions was used. This table is illustrated in Fig. 11. It was made by the Traylor Engineering Company of New York City, and is similar to other well-known makes, which include the Wilfley, Overstrom, Deister, Butchart, and others. The material fed to the table in the test runs was effectively separated into fine pyrite, containing on the average run more than 40 per cent sulphur, and fine coal which might be added to the coal obtained from the jigs. As a rule, the handling of quantities of such fine coal presents some difficulty because of the problem of removing the water from it after washing.

15. *Losses.*—The tests indicate either 81 or 87 per cent recovery of the pyrite as shown by sulphur analysis, and therefore show a loss of 19 and 13 per cent, respectively. This seems to be a satisfactory metallurgical recovery for such a washing process, especially since the effort has been to employ the simplest sort of machinery.

Probably 5 to 20 per cent of the coal in the smaller sizes will be lost during the process, largely by passing off as sludge in the water. Analyses indicate that this sludge is too impure to be used as coal. Not only is it very fine, but much of the clay or shale impurity which is intermixed with the coal has softened under the influence of the water and passes off with the sludge.

16. *Water Supply.*—In any wet concentration process using jigs and concentrating tables, the question of an adequate supply of water is important. Water is used in spraying the rock in the breaker for the purpose of keeping down dust; it is used in the disintegrating screen to assist in the cleaning; and from this point the material under  $1\frac{1}{2}$  inches in size is practically flowing in a stream of water. The jig and concentrating table both require water, for the feed, for

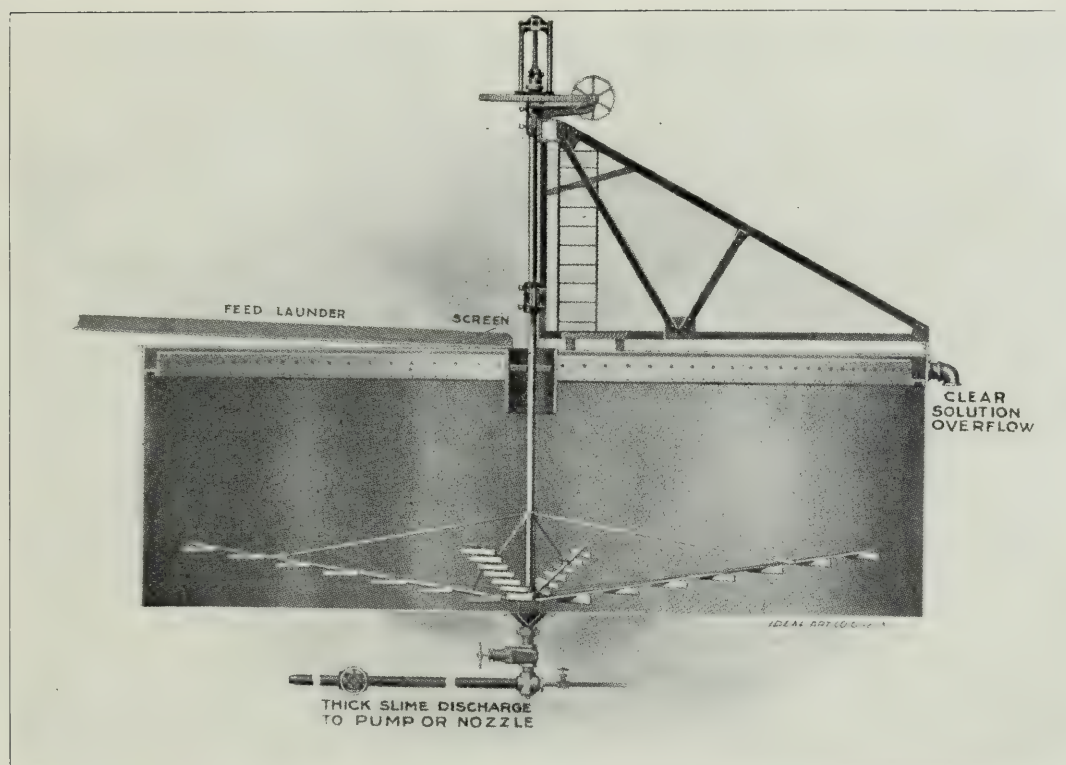


FIG. 12. THE DORR CONTINUOUS THICKENER (PATENTED). INSTALLED  
IN STEEL TANK





the separating process, and for carrying away the separated products. By the use of perforated elevators and draining bins it is possible to recover all the water draining from the products in a central pond or sump and to use it over and over by pumping. The sediment or sludge in the water consists largely of fine coal and clay, since the pyrite is too heavy to pass off with the water except in the smallest sizes. The settling pond or sump common at Illinois coal washeries can be replaced to good advantage by a large round settling tank 20 or 30 feet in diameter equipped with a uniformly horizontal rim over which the water may flow.

The sludge water from the plant should enter this tank at the center and under the surface of the water. Passing toward the rim of the tank, the sludge will settle to the bottom and the water, sufficiently clean to be reused, will overflow the rim and may be directed into a small sump from which it may be pumped back to the plant. The tank should be equipped with a steeply sloping bottom so that the accumulating sludge may be easily removed. A settling tank of similar character, which has been used for many industrial purposes, is the Dorr settling tank illustrated in Fig. 12. The importance of fairly clean water in the operation of a plant of this kind may be readily understood since water used several times without settlement of the sludge often contains as much as 3 per cent of solids in suspension. Such water, draining from the washed pyrite, will contaminate it by depositing solid material on its surface and will thus lower the sulphur percentage of a product otherwise satisfactory.

17. *Design of Plant.*—Fig. 13 shows a suggested plan of a pyrite washing plant based on the flow sheet presented as Fig. 4. Fig. 14 is a side elevation of the same plant. By following the arrows indicating direction of flow of the material, the operation of the plant may be readily understood.

The bins, framing, and general construction material may be of wood or steel. Since the acid water formed through contact with pyrite has a corrosive action in contact with steel, it is suggested that at least the bin linings be of wood. At present, general wooden construction should be much cheaper than steel.

In the design presented, the site has been assumed as level. If a sloping, or side hill, site is available, some expense for elevators may be avoided. In cases where elevators are required, they should



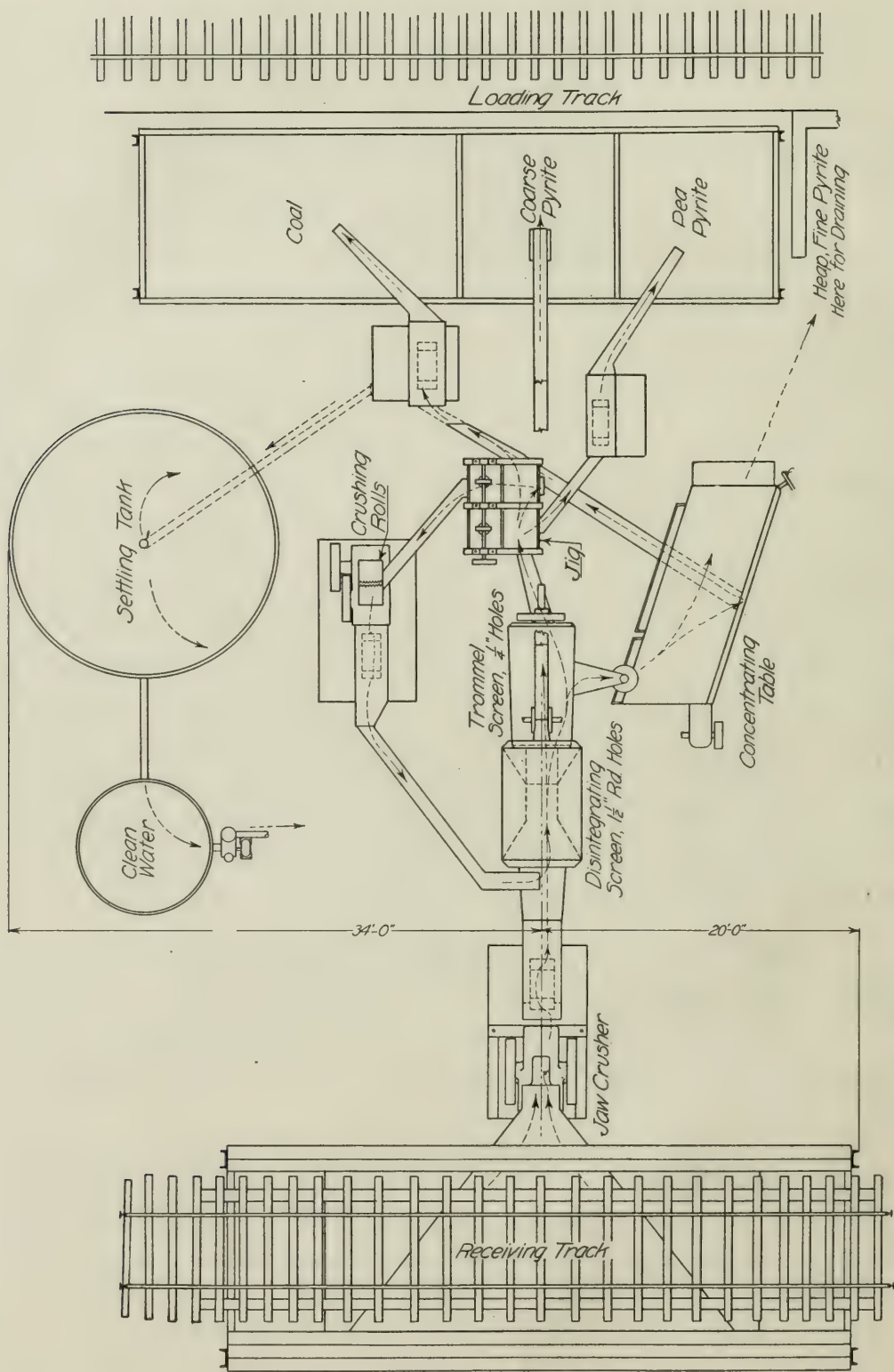


FIG. 13. PLAN OF PROPOSED PYRITE WASHING PLANT

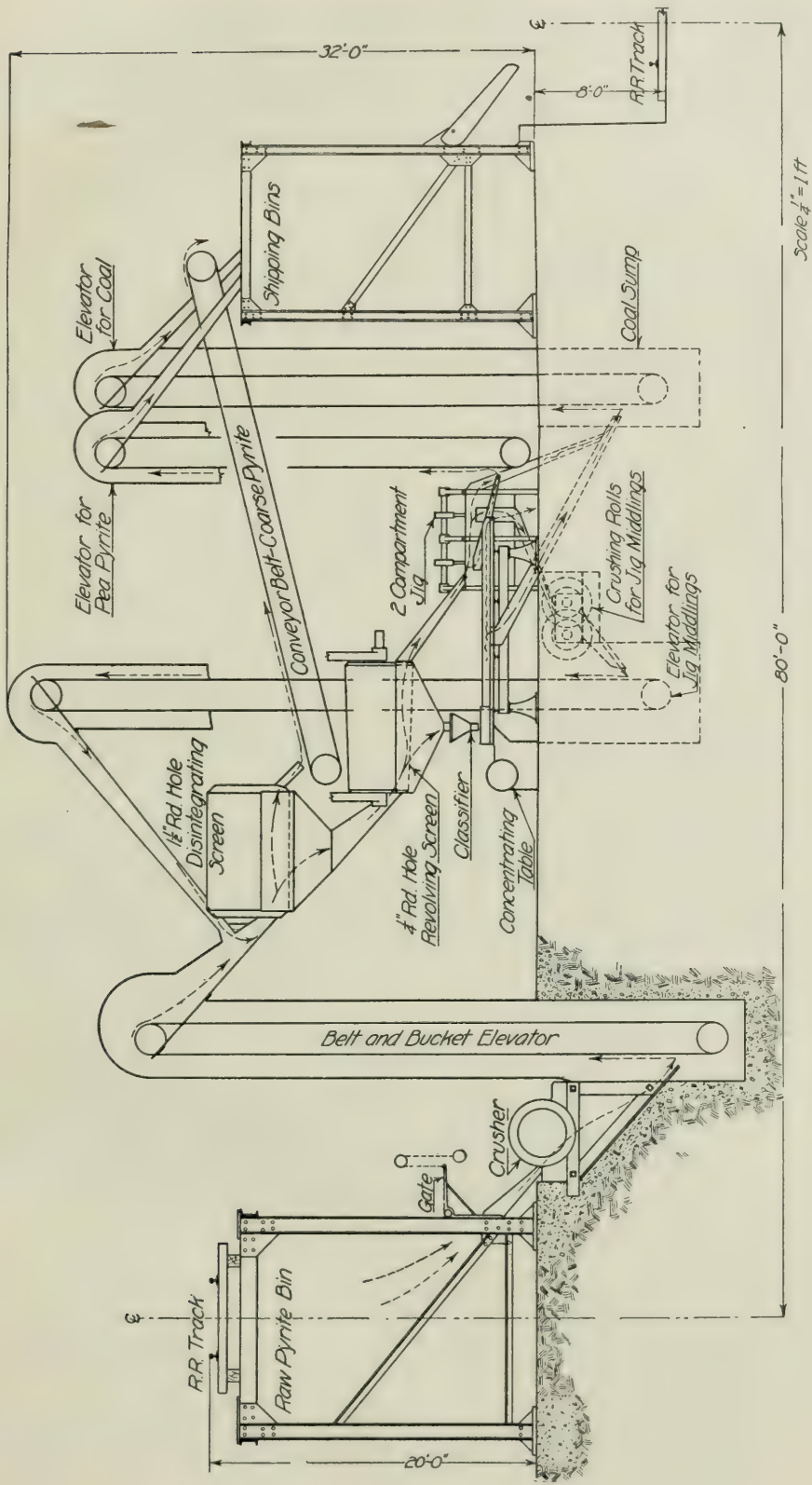


FIG. 14. SIDE ELEVATION OF PROPOSED PYRITE WASHING PLANT



be of the rubber belt and steel bucket type, since these are cheaper to install and, for the purpose intended, will last longer than the all-steel types. In a small plant, bins may be omitted, and the cleaned products may be allowed to drain on to a concrete floor, as indicated in Fig. 13 for fine pyrite.

18. *Cost of Plant.*—The design outlined herein was submitted to Allis-Chalmers Manufacturing Company, Milwaukee, Wisconsin on July 6, 1917, with the request that they submit an estimate of the cost. In their reply they suggest that a disintegrating screen somewhat smaller than the one illustrated in Fig. 9 should be satisfactory. With this exception, their estimates cover the design and the arrangement of plant as outlined in Figs. 13 and 14. The list of equipment and the estimates of cost as submitted by Allis-Chalmers Manufacturing Company are as follows:

“1—Blake Crusher, 15x9.

1—Disintegrating screen or trommel 48 inches diameter by 8 feet long having punched steel covering with  $1\frac{1}{2}$  inch openings and provided with three angle iron lifters equally spaced inside the screen.

1—Trommel 48 inches diameter by 8 feet long provided with punched steel covering having  $\frac{1}{4}$  inch round holes.

1—Two-Compartment Harz Jig with compartments about 24 inches by 36 inches to be complete including iron work, wood work and screens.

1—Set 18 inches by 10 inches Economic crushing rolls.

1—Belt and bucket elevator about 35 feet long between centers and to be provided with top and bottom shafts, pulleys, bearings, driving shaft, driving gear—16-inch malleable elevator buckets with bolts and 18-inch 8-ply rubber belt (other elevators or conveyors extra as needed).

1—Overstrom concentrating table.

1—Lot of shafting pulleys, boxes, and belting for driving the previously described machinery in accordance with plans to be made.

1—Lot of piping, valves, and fittings for water supply to the various machines specified but not including connections outside of building.

1—Set of drawings of plan and elevator showing the general arrangement of the plant will be provided with the machinery.

“The price of the machinery as outlined would be \$5,600.00 f. o. b. cars at the factory, approximate weight 42,000 lb.

“Shipment could be made in about 60 days from receipt of order.

“The foregoing covers what would be practically the standard equipment for most of the plants, and the following details might vary with different installations.

“1—40 H. P. type A N. Ind. motor, 60 cycle, 3 phase, 440 V. 865  
R. P. M. for driving plant,  
Approximate weight .....2,000 lb.  
Price .....\$550.00

1—Water tank for collecting waste water from plant for reuse.  
Tank would be 20 inches diameter with 10-foot staves made of  
3-inch lumber and provided with overflow launder. The capacity  
of this tank would be about 20,000 gals.  
Approximate weight .....8,000 lb.  
Price .....\$300.00

“This tank may seem a little large but the larger it is made the better will be the settling action which removes any fine coal or gritty material from the overflow.

“To return the water from this tank to the supply tank there will be required a pump, but probably in every case this pump would have to be changed to suit the conditions of the head and the quantity of water to be returned. It is assumed for average conditions that the pump can be motor driven and that a centrifugal pump will be most satisfactory for this service; also that a capacity of about 100 gals. per minute would be sufficient and that the head would be about 50 feet.

“2-inch type ‘S’ centrifugal pump with direct connected motor, mount on base plate. The motor would be 5 H. P. Ind. motor, 60 cycle, 3 phase, 440 volt.

The weight of this pump would be.....1,100 lb.  
Price of same.....\$325.00

“No suction or discharge piping for this pump is included as this will have to be made to suit the conditions in each case.

“No clear water supply tank for the plant or pump for same is included.

“These prices are based on the present conditions only.”



19. *Summary of Capital Costs.*—In the following summary the initial capital requirements are presented for a pyrite recovery plant having a capacity of 50 tons per 8-hour day as described in the foregoing paragraphs of this circular.

|                                   |           |
|-----------------------------------|-----------|
| General Machinery at Factory..... | \$5600.00 |
| 40 H. P. Motor.....               | 550.00    |
| Water Settling Tank.....          | 300.00    |
| Water Pump .....                  | 325.00    |
|                                   | <hr/>     |
|                                   | \$7775.00 |

or approximately \$8000.00 delivered.

If it is assumed that the installation of the machinery will cost fifty per cent of its original cost, or about \$4000.00, including foundations, and that the building and bins will cost about \$6000.00 (this will vary as to location and completeness of plant desired), the total cost of a complete pyrite plant capable of treating 50 tons of crude hand-picked pyrite per 8-hour day will be about \$18000.00.

## APPENDIX

20. *Analysis of Pyrite Ores for Sulphur Content.*—The sulphur analyses listed in this circular were made according to the method described in Low's "Technical Methods of Ore Analysis," page 239, with the exception that it has been possible to shorten the general procedure somewhat since the material contains no sulphide or impurity requiring removal. It is a simple, accurate, and practical method for determining sulphur in large percentages, such as are found in pyrite. It is presented in the following paragraphs:\*

"Treat 0.5 gram of the ore in an 8-ounce flask with 10 cubic centimeters of strong nitric acid. Heat very gently until the red fumes have somewhat abated, and then add potassium chlorate in small portions at a time (say 0.2 to 0.3 gram), until any free sulphur that has separated is entirely oxidized and dissolved. The acid should not be boiled violently, as this would unnecessarily weaken it. On the other hand, it is best not to allow it simply to simmer, as the explosive gases from the decomposing chlorate may then collect in the flask and produce annoying, although not dangerous, explosions. When the sulphur has entirely disappeared, the solution should be boiled to complete dryness. This operation may be hastened by manipulating the flask over a free flame. After cooling, add 5 cubic centimeters of strong hydrochloric acid. This should be done cautiously to avoid a too violent reaction with the undecomposed potassium chlorate that may be present. If iron oxide, etc., still remains undissolved, gently heat the hydrochloric acid mixture until solution is as complete as possible, adding more acid if necessary. Finally, boil to dryness, then add 5 cubic centimeters more of the hydrochloric acid and again boil to dryness. This is to decompose nitrates and expel all nitric acid. Take up once more in 5 cubic centimeters of the hydrochloric acid and dilute with about 100 cubic centimeters of cold water. (If the solution is hot when made alkaline with ammonia, some basic ferric sulphate is liable to separate.) Make alkaline with ammonia. Heat to boiling, allow the ferric hydroxide, etc., to settle, and then filter and wash very thoroughly with hot water, receiving the filtrate in a 600 cubic centimeter Erlenmeyer flask.

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\*Attention is directed to the so-called sodium peroxide method of sulphur analysis involving the use of a combustion bomb. This is perhaps the quickest and is also an accurate method for the analysis of sulphur found in ores. For a description see "Sodium Peroxide in Certain Quantitative Processes," Jour. Amer. Chem. Soc., Vol. 30, 1908.



“Add a drop of phenolphthalein solution as indicator, make just acid with strong hydrochloric acid, and then add 4 cubic centimeters in excess. Dilute the solution to about 400 cubic centimeters with hot water, heat to boiling and precipitate the sulphur with barium chloride.

“After adding the barium chloride, as described, to the boiling solution, allow the mixture to stand, hot, until the liquid above the precipitate has become perfectly clear—perhaps an hour. Filter through a double 11 centimeter filter. No appreciable amount of barium sulphate should run through. Unless, however, the filter appears practically clear, always filter a second time, which will usually suffice. Wash the precipitate ten times with hot water. Transfer the moist filter and precipitate to a clean smooth ‘annealing-cup’ and ignite, with free access of air, over a Bunsen burner, or in a muffle, at a gentle heat. A high heat, such as that of a blast lamp, is neither necessary or desirable. The ignited barium sulphate should be perfectly white. Cool in dessicator, transfer to the scale-pan by tapping and brushing with a camel’s hair brush, and weigh. Multiply the weight of the barium sulphate by 0.1373 to obtain the weight of the sulphur.

“A platinum or porcelain crucible may, of course, be used for the ignition of the barium sulphate instead of an annealing-cup.

“It is best to run a blank, once for all, with all the reagents employed, and always deduct for any sulphur thus found.”

21. *Additional Directions and Precautions.*—The sulphur percentage in barium sulphate is 13.73.

The whole operation of solution and evaporation can best be carried on in a round-bottom flask over a free flame.

It is important to keep the flask in motion constantly to avoid cracking, and after evaporation not to set the flask down on a solid substance (as a desk) until it has cooled somewhat.

Accurate results depend largely upon the complete removal of the nitric acid through boiling, and for this reason the operation of evaporation is repeated.

The filtering of the solution to remove the ferric hydroxide is hastened by allowing the precipitate to settle and by decanting the clear solution.

If the solution is allowed to stand for several hours, or over night, after adding the barium chloride, heating is usually not necessary.

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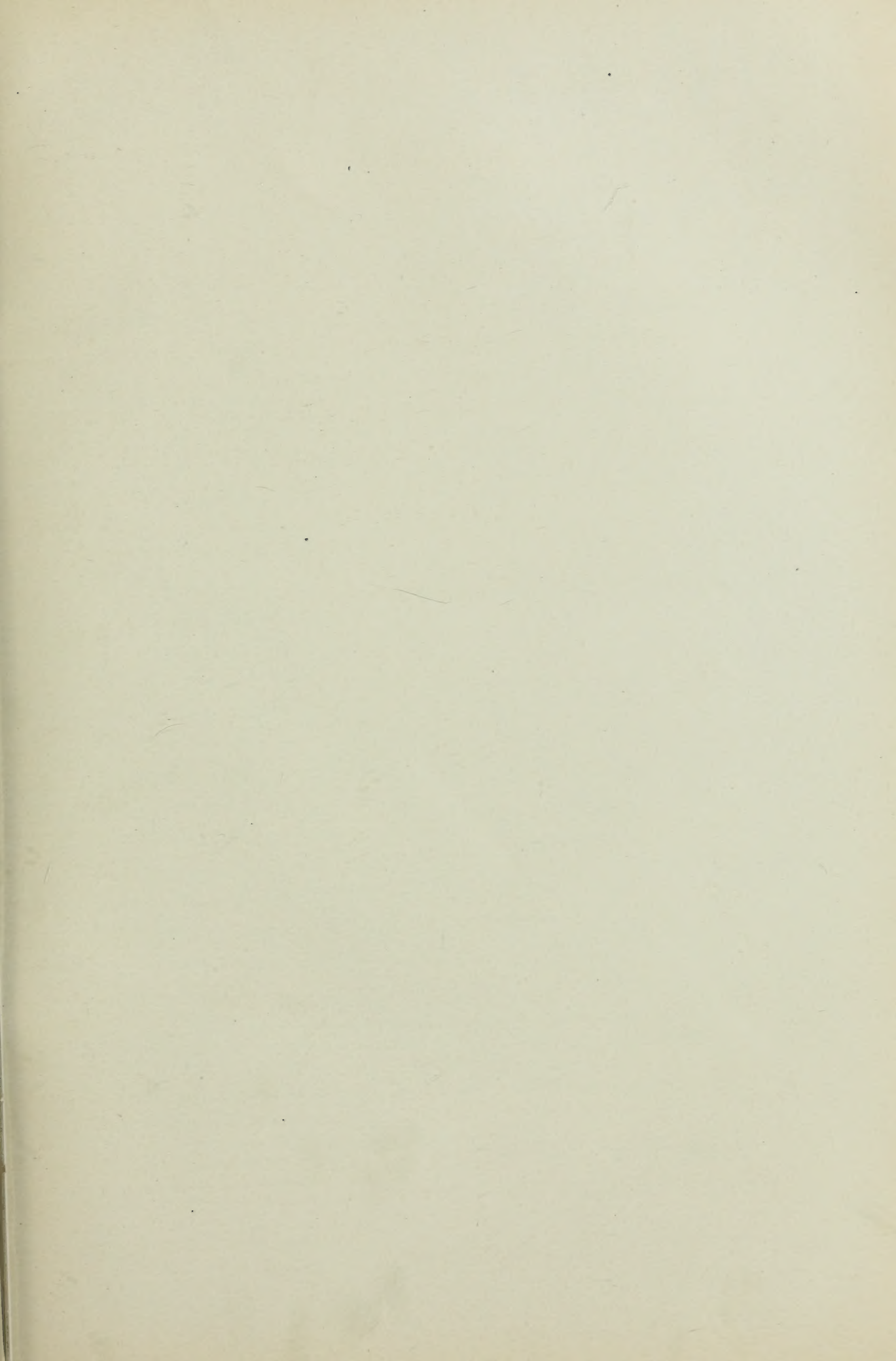
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